(Reaffirmed 2018)

भारतीय मानक

लुगदी के लिए परीक्षण पद्धति

भाग 13 तांबा सामग्री ज्ञात करना

(पहला पुनरीक्षण)

Indian Standard

METHODS OF TEST FOR PULP

PART 13 DETERMINATION OF COPPER CONTENT

(First Revision)

ICS 676.1 : 676.014.2 : [546.56]

B U R E A U O F I N D I A N S T A N D A R D S
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NEW DELHI 110002

September 2013 **Price Group 2**

Paper and Its Products Sectional Committee, CHD 15

FOREWORD

This Indian Standard (Part 13) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Paper and Its Products Sectional Committee had been approved by the Chemical Division Council.

Copper, iron and manganese, if present in pulp in more than traces, not only discolour it but may also affect its dyeing properties. It is, therefore, essential to know the quantities of each of these in the pulp.

This standard which was prepared mainly based on ISO/R 778-1968 'Pulps — Determination of copper' first published in 1975. As this version was very old, decision has been taken for its revision to update the standard. It is for information that International Organization for Standardization (ISO) has published a standard ISO 12830 : 2011 'Paper, board and pulp — Determination of acid-soluble magnesium, calcium, manganese, iron, copper, sodium and potassium'. As this ISO method is for paper, board and pulp and the IS 6213 (Part 13) prescribes method of test for pulp only, the Committee also decided to adopt ISO 12830 : 2011 as dual number standard in future.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR PULP

PART 13 DETERMINATION OF COPPER CONTENT

(First Revision)

1 SCOPE

This standard (Part 13) prescribes the method for the determination of copper content of pulp.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
6213 (Part 7) : 1971	Methods of test for pulp: Part 7 Ash content in pulp

3 PRINCIPLE OF THE METHOD

The pulp is ashed and the ash is dissolved in hydrochloric acid. The copper is extracted with sodium diethyl dithiocarbamate in carbon tetrachloride. The concentration of copper is determined colorimetrically by measuring the optical density at 435 nm. Disturbing ions are masked with disodium salt of ethylene diamine tetraacetic acid.

4 QUALITY OF REAGENTS

Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070), freshly boiled and cooled, shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of test.

5 REAGENTS

5.1 Carbon Tetrachloride

5.2 Ammonia, concentrated solution (r.d. 0 : 9 1).

5.3 Hydrochloric Acid — 6 M approximately. Dilute 500 ml of hydrochloric acid (r.d. 1.19) with 600 ml of water.

5.4 Solution of Disodium Salt of Ethylene Diamine Tetra-acetic Acid (EDTA) — 5 percent. Dissolve 50 g of the disodium salt of ethylene diamine tetra-acetic acid ($C_{10}H_{14}O_8N_2\ Na_2.\ 2H_2O$) in water and dilute to 1 litre**.** Keep the solution in a polyethylene bottle.

5.5 Sodium Diethyl Dithiocarbamate Solution — Dissolve 0.1 g of sodium diethyl dithiocarbamate [$(C_2H_5)_2N$ CSSNa. 3 H_2O] in 100 ml of water. Filter any insoluble matter present and store the solution in a dark bottle. The solution can be kept unchanged for about one week.

5.6 Standard Copper Solution — It contains 0.1 mg of copper per millilitre. Dissolve 0.100 g of pure, electrolytic copper metal in the smallest possible quantity of nitric acid (r.d. 1.4). Boil in order to expel nitrous fumes, allow to cool, transfer quantitatively to a 1 litre volumetric flask and dilute to the mark with water.

5.7 Phenolphthalein Solution — Dissolve 50 mg of phenolphthalein ($C_{20}H_{14}O_4$) in 50 ml of ethanol (C_2H_5OH), and dilute with 50 ml of water.

6 APPARATUS

6.1 Dishes, of quartz.

NOTE — Platinum or porcelain dishes should not be used.

6.2 Weighing Balance, of suitable capacity having least count 0.01 mg.

6.3 Spectrophotometer or Filter Colorimeter

6.4 Cells

7 CALIBRATION

7.1 Dilute the standard copper solution ten times so that 1 ml corresponds to 0.01 mg of copper, With a pipette take aliquots of 2.0, 5.0, 7.0 and 10.0 ml of the diluted solution and place them into 250-ml separating funnels. Use a fifth funnel for the preparation of a reference solution. Add 20 ml of hydrochloric acid, 10 ml of EDTA solution and five drops of phenolphthalein solution to each funnel. Neutralize the solution with concentrated solution of ammonia to a faint pink colour and cool to room temperature. Add 5 ml of sodium diethyl dithiocarbamate solution and, using a pipette, 20.0 ml of carbon tetrachloride. Shake

the funnel vigorously for at least 3 min and then leave the phases to separate.

7.2 Filter the carbon tetrachloride phase (the lower phase) through a cotton plug inserted in the outlet of the separating funnel, into a cell, discarding the first 5 ml. Apply the lid of the cell and immediately measure the optical density at 435 nm of the carbon tetrachloride phase of the funnel with the copper-free solution as a reference.

NOTES

1 Avoid exposure of the coloured carbon tetrachloride solutions to direct sunlight. The copper concentrations of the coloured carbon tetrachloride solutions would be 1.0, 2.5, 3.5 and 5.0 mg of copper per litre, respectively. Plot on a graph the absorbance values, divided by the length of the cell, against the copper concentration and check that the points lie in a straight line going through the origin.

2 The copper content of the distilled water should be very low. If the absorbance of the reference solution compared to water exceeds 0.05, the water should be purified, for example, by ion exchange.

8 PROCEDURE

8.1 Preparation of Test Sample

Tear the air-dry sample into pieces of a suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred. Weigh about 10 g of pulp (or 5 g for copper content above 10 mg/kg) to the nearest 0.01 g. At the same time weigh out a separate sample for the determination of dry matter content.

8.2 Determination

8.2.1 Ash the test sample as described in IS 6213 (Part 7) using a clean quartz dish. Burners of brass or other material containing copper should not be used.

8.2.2 In order to obtain a reagent blank, take another clean dish of the same type as used for the ashing of the test piece and treat as described below:

Add to each dish 5 ml of 6 m hydrochloric acid and evaporate to dryness on a steam-bath. Repeat this once and then treat the residue with another portion of 5 ml of hydrochloric acid and heat for 5 min on the steam-bath. Transfer the contents of the dishes to separating funnels with the aid of water. Add to the insoluble residue in each dish a further 5 ml of hydrochloric acid and heat on the steam-bath. Transfer this last portion to the main quantity in the separating funnel with the aid of water.

Add 10 ml of EDTA solution and 5 drops of phenolphthalein solution to each funnel. Neutralize the solution with concentrated solution of ammonia to a faint pink colour and cool to room temperature. Add 5 ml of sodium diethyl dithiocarbamate solution and, using a pipette, 20.0 ml of carbon tetrachloride. Shake the funnels vigorously for at least 3 min and then leave the phases to separate. Filter into a cell the carbon tetrachloride phase (the lower phase), through a cotton plug inserted in the outlet of the separating funnel, discarding the first 5 ml. Apply the lid of the cell and immediately measure the optical density at 435 nm with the reagent blank as a reference. Divide the reading by the length of the cell.

NOTES

1 Avoid exposure of the coloured carbon tetrachloride solutions to direct sunlight.

2 *Cleaning of dishes* — Wash the dish thoroughly, remove any spots in dishes rubbing with fine sand. Boil the dish four times with 6 M hydrochloric acid and avoid any possible contact with copper.

9 CALCULATIONS

Carry out two determinations and calculate as follows:

$$\text{Copper content, in mg/kg (parts per million)} = \frac{a\,v}{m}$$

where

a = amount of copper obtained from the absorbance values and the calibration curve, expressed, in milligrams per litre of coloured solution;

v = volume of the coloured solution, in ml; and

m = mass of pulp, calculated on an oven-dry basis, in g.

Report the result as the mean of the two determinations, in milligrams per kilogram (parts per million), to the first decimal place.

NOTE — The precision of the method as such is high. Thus, the difference between results of duplicate tests is usually attributable to uneven distribution of the trace metal in the pulp.

10 TEST REPORT

The test report should state the results obtained and, moreover, indicate all the conditions of the test, any details of procedure regarded as optional or not laid down in this standard and any incidents that may have affected the results.

ANNEX A
(*Foreword*)

COMMITTEE COMPOSITION

Paper and Its Products Sectional Committee, CHD 15

Organization	*Representative(s)*
Central Pulp and Paper Research Institute, Saharanpur	Dr R. M. Mathur (***Chairman***)
All India Federation of Master Printers, New Delhi	Shri P. Chander Shri V. Ravi (*Alternate*)
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